An Electrostatic Precipitator for Preparative Gas-Liquid Chromatography

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ABSTRACT

The effect of the operating variables of electrostatic precipitators on the recovery and structure of methyl esters and related aerosol forming compounds collected in preparative gas-liquid chromatography was studied.

Aerosol formation was prevented by AC or DC voltages of 5000 to 12000 volts. AC was more effective than DC but caused changes in structure which were detectable by both thin-layer and gas-liquid chromatographic methods of analysis.

An apparatus of simple construction and operation was designed for the collection of methyl esters and its use demonstrated with several model compounds.

INTRODUCTION

Electrostatic precipitation (1) was first applied to preparative gas-liquid chromatography (GLC) for the collection of samples by Atkinson and Tuey (2). In the course of the development of this technique, modifications in the apparatus have been described by Kratz et al. (3), Thompson (4), Snelson (5) and Ross et al. (6). These workers have shown that aerosol formation which is the main factor resulting in low recoveries in preparative GLC can be broken by voltages of greater than 3400 volts.

Both AC and DC voltages have been employed in electrostatic precipitators and no specific recommendation regarding their use has been made except that AC has been reported to be more effective than DC in the prevention of aerosol formation.

Since high voltages are known to cause changes in the structures of organic compounds (7), we made a study of the operating variables of electrostatic precipitators for preparative GLC and designed a simple apparatus for the collection of methyl esters and related compounds.

EXPERIMENTAL

Materials

Hexadecane >99% purity was obtained from Matheson, Coleman and Bell. Highly purified (>99%) stearic acid, methyl oleate, methyl linoleate, methyl linolenate, methyl palmitate, palmityl alcohol, methyl laurate, capryl alcohol and caprylic acid were obtained from The Hormel Institute, Austin, Minnesota.

Apparatus

The gas chromatograph used throughout this work was an F & M Model 500 equipped with a thermal conductivity detector and 7 ft × ¼ in. column packed with 10% (w/w) ethylene glycol succinate polymer on 100–120 mesh Chromosorb P. Helium was used as the carrier gas at 75 ml/min. The column and block were operated at temperatures of 185 and 250C, respectively.

The electrostatic precipitator designed for this study is shown in Figure 1.

A schematic diagram of the apparatus is shown in Figure 2. It has two main features: 1) It is of very simple construction. 2) It is simple to operate as it can be quickly and easily disassembled. Some of the construction features of the apparatus are as follows: The inner electrode consists of a 35 cm × 1.5 mm *o.d.* steel wire inside a glass tube. The outer electrode consists of a 1/16 in. sheet of aluminum foil (28 × 13 cm) and it is wrapped around a glass tube (44 × 0.9 cm) and fastened by plastic tape. The distance between the two

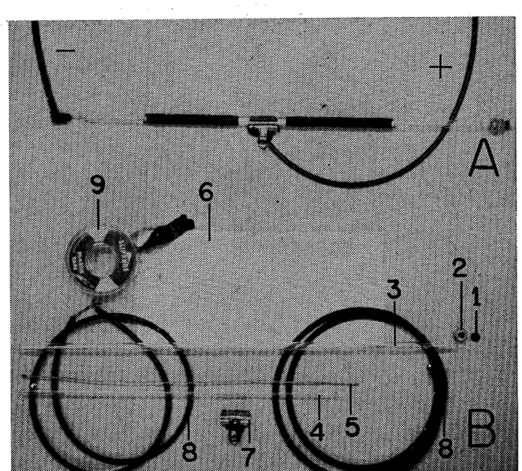

FIG. 1. *A*, assembled electrostatic precipitator; *B*, the component parts of A; *1*, silicone rubber disk; *2*, nut; *3*, outer glass tube; *4*, inner glass tube; *5*, inner electrode; *6*, aluminum sheet; *7* clamp; *8*, insulated wires; *9*, plastic tape.

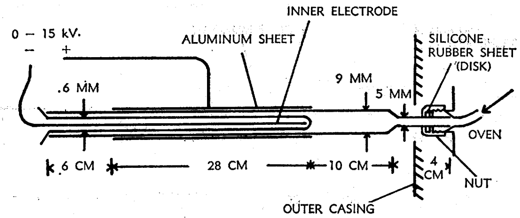

FIG. 2. Schematic diagram of electrostatic precipitator.

glass tubes is narrow (0.5 mm) to make the passageway for the aerosol narrow and at the same time provide a large surface area of electrode per volume of the passageway. The steel wire can be readily withdrawn from the tube within which it is placed and the wire from the transformed to the outer electrode can also be readily removed to permit the quick (few seconds) replacement of another collection unit A (Fig. 1).

For the collection of samples the outer tube of the unit is inserted through the septum of the outlet of the gas chromatograph until it comes into contact with the end of the column.

Experiments were carried out with both alternating and direct current. For the alternating current experiments the precipitator was connected to an AC transformer with an input of 115 v and an output of 15 kv (30 ma). The input voltage for the high voltage transformer was regulated by means of a variable transformer which made it possible to produce voltages from zero to 15,000.

DC voltages were obtained by means of a DC power supply with an input of 118 AC and an output of 15 kv at 1.5 ma DC (Model No. HV150-152M; Plastic Capacitors Inc., Chicago, Ill.). It was also connected to a variable transformer to provide a range of voltages.

RESULTS

In the first experiments the voltage required to prevent aerosol formation was determined by observation of the smoke or fog which emerged from the outlet of the precipitator when it was alternately turned on and off. The results for a variety of compounds of different structures are shown in Table I. In accordance with the observation of Kratz et al. (3) AC voltage was more effective in the prevention of aerosol formation than DC voltage.

Recovery Experiments

In order to eliminate spurious results from column bleed the amount of recovery was measured by analytical GLC using an internal standard. In this procedure the amount of sample collected was determined by dissolving it in a known volume of a standard solution of the compound used as an internal standard, and comparing the peak areas of the standard with that of the collected substance. The results of the recovery of several methyl esters via the electrostatic precipitation is compared to gradient cooling condensation which is superior to cold trap precipitation in Table II. The results (Table II) show that the recoveries of aerosol-forming compounds average about 96%. These experiments illustrated the application of the apparatus to analytical separations obtained with the conventional ¼ in. column. Essentially 100% recoveries may be obtained with large diameter preparative columns because the actual loss of material appears to be about the same, regardless of the amount of sample applied to the column.

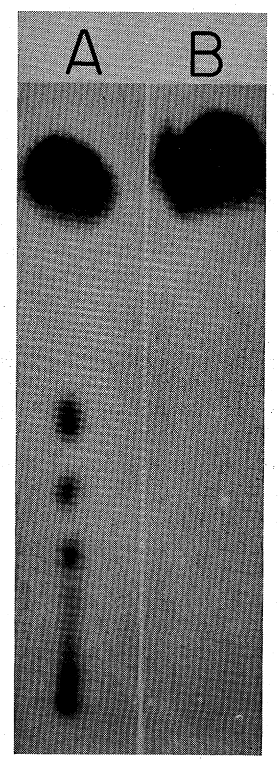

FIG. 3. Thin-layer chromatoplate, showing methyl oleate, treated with high voltage. *A*, the effect of AC high voltage; *B*, the effect of DC high voltage. Coating: Silica gel G, developed in a mixture of petroleum ether (30–60C), ethyl ether (90:10). Charred at 180C after spraying with 70% sulfuric acid (v/v), saturated with potassium dichromate.

TABLE I

Precipitation of Aerosol-Forming Compounds

Compound	AC voltage	DC voltage
Stearic acid	5500	12000
Methyl oleate	5500	8200
Methyl linoleate	5500	9500
Methyl linolenate	6800	11000
Hexadecane	6500	10200
Methyl palmitate	6100	12300
Palmityl alcohol	5700	7500
Methyl laurate	6100	10200
Capryl alcohol	5500	8200
Caprylic acid	5500	10600

Structural Alteration of Compounds

When direct current was employed, aerosol formation could be prevented without any evidence of structural alteration to the compounds.

Alternating current caused changes with all compounds. Changes could be detected with some compounds at voltages below that required to break aerosol formation. The alteration in structure was determined mainly by thin-layer chromatography (TLC). The chromatoplate in Figure 3 shows the analysis of methyl oleate collected via AC and DC voltages. In no case could alternating current be employed without evidence of some structural alteration of the collected compounds. In addition to nonvolatile products, gases such as methane, ethane are generally formed. The mechanism and products of discharge reactions of the type encountered in electrostatic precipitators using AC current will be described in more detail in a separate report by the authors (8).

DISCUSSION

Low recoveries of compounds encountered in preparative GLC because of aerosol formation may be greatly improved by the use of electrostatic precipitation techniques. With long-chain methyl esters no other devices are required to attain recoveries of the order of 95%, and when DC voltages are employed no alteration in the structure of the collected samples occurs. AC voltage caused changes in structure of methyl oleate via discharge reactions. The structural changes in this and related compounds may be readily detected by TLC. Polar compounds are formed as well as short-chain hydrocarbons which may be detected by GLC as demonstrated in further work from this laboratory (8).

TABLE II

Recovery of Methyl Esters via Electrostatic Precipitation Technique

Compound	Remark	% Recovery by only condensation	% Recovery by condensation and electrostatic precipitation
1 μl Methyl caprylate	No visible aerosol	87.0	90.1
1 μl Methyl oleate	Aerosol	88.6	96.1
1 μl Methyl arachidonate	Aerosol	79.1	96.3

In addition to the use of DC voltage there are certain other features which should be considered in the design of a precipitator for preparative GLC. These may be enumerated as follows:

1. Since high voltages are employed, the apparatus should be constructed to minimize the hazard of electrical shock.
2. The unit should permit the collection of multiples of samples in a short period of time.
3. The design should attain a gradient cooling of the emerging vapors before they enter the electrical field to decrease the amount and stability of the aerosol.
4. The apparatus should be simple to permit fast assembly and cleaning.
5. Finally, it should be pointed out that since discharge reactions generally give rise to polymers and highly volatile products (among others), GLC is not a good method for the detection of structural changes unless conditions are used to detect these types of compounds.

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